INTRODUCTION

TO

SUGAR ANALYSIS.

NOTES ON

Lectures and Laboratory Manipulation.

BY

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BOSTON:
PRINTED FOR THE AUTHOR.
1898.

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EARLIER METHODS.

Difficulties in determining cane-sugar by the usual analytical methods. Four processes of limited application not meeting commercial requirements:

(1) *Fermentation by Yeast.*— (a) By action of the invertase; $C_{12} H_{22} O_{11} + H_2 O = 2 C_6 H_{12} O_6$. (b) By fermentation; $C_6 H_{12} O_6 = 2 C O_2 + 2 C_2 H_5 O H$. Objections — Long time required. Action of foreign ferments.

(2) *Inversion and Titration with Fehling Solution.*— Treatment with hydrochloric acid. Preparation and manipulation of Fehling solution in volumetric method. $Cu_2 O$ precipitated. Conditions affecting accuracy. Time of boiling—dilution. Soxhlet's modification. Gravimetric methods — $Cu O$ or $Cu_2 O$ weighed (O'Sullivan), Cu (Allihn), Cu by electrolysis (Wiley).

(3) *Specific Gravity at standard temperature*, 17.5° C. — Balling-Brix hydrometers. Only accurate where soluble impurity is small. Valuable in commercial work in connection with other methods.

(4) *Extraction by Alcohol.*— Accurate, but only applicable to substances containing small quantities of sugar. Sugar weighed directly.

OPTICAL ANALYSIS.

General Principles.— Biot, 1815, discovered action of solutions of sugar and certain other organic substances on polarized light rays. Double refraction caused by some crystals. Iceland spar. Suppression of one member of doubly refracted ray by Nicol prism. Passage of light through two Nicol prisms in succession. (a) edges of end

faces parallel; (b) edges at right angles. *Characteristics of Polarized Light Beam, Essential to its Use in Optical Analysis* (as shown by its passage through two Nicol prisms in succession). (1) A beam of given intensity can pass only at a *definite position* of the prisms relative to each other. Interpretation by wave theory. Diagrams: ordinary light beam, polarized beam. Conclusion of Theory: That a polarized light beam can be considered as having its vibrations in one plane ("plane of polarization") whose direction in space bears a constant relation to the principal section of the Nicol prism. (2) Rotation of beam by sugar solution. How shown.

Angle of rotation (a) proportional to concentration (c), distance traversed through solution (l), and varies with the color of the light ray, the proportional effect on each color being the same as that caused by the dispersion of the solution. *The angular degrees of rotation of the polarized ray of standard color caused by one gram of the optically active substance dissolved in each cubic centimeter of solution in a column one decimeter long* are taken as a measure of the *Specific-Rotatory Power* (a) of that substance. The Fundamental Formula expressing rotation of the polarized ray by any optically-active substance: $a = alc$ when (a) is angle of rotation in *degrees*, (l) = length of column in *decimeters* and (c) = concentration of the solution of (w) *grams* of optically-active substance in (v) *cubic centimeters* of solution.

$c = \dfrac{w}{v} \therefore a = \dfrac{alw}{v}$ and $a = \dfrac{av}{lw}$. When (c) is expressed as

per cent of substance in solution $\left(\dfrac{p}{100}\right)$ the density (d) must

be known, $\dfrac{w}{v} = \dfrac{pd}{100} \therefore a = \dfrac{100a}{lpd}$. [Note carefully the distinction between grams in 100 *cubic centimeters* and grams in 100 *grams* of solution.] For liquids not requiring solvents,

$\dfrac{w}{v} = \dfrac{1}{d}$ where d = density of liquids, $a = \dfrac{a}{ld}$. For solid

sections, as quartz; $a = \dfrac{a}{l}$, $l = 1$ millimeter. When standard yellow light (D) ray of spectrum) is used, a is expressed

$[a]_D$; when "mean yellow" light is used (measured by observation of its complementary "transition-tint"), $[a]_J$. Constancy of a affected in some cases by variations in temperature, concentration, and nature of solvent. a of cane-sugar practically constant under ordinary conditions of laboratory practice. Standard temperature, 17.5°C (more recently, 20°).

APPLICATION OF PRINCIPLES TO SUGAR ANALYSIS.

w' = weight of sample. w = weight of sugar in sample

$$\therefore \frac{w}{w'} = \% \text{ sugar } w = \frac{av}{al}, \ \% = \frac{av}{alw'}.$$

POLARISCOPES.

Essential parts; polarizer, tube for solution, rotating analyzer with scale for measuring angle of rotation, optical device for determining exact position of plane of polarization relative to the analyser, source of light necessarily monochromatic. Early polariscopes used day or lamp light getting monochromatic effect by "transition-tint" plate, giving $[a]_J$. Now, all polariscopes in common use employ sodium flame filtered through potash bichromate. This gives light closely approximating to D ray, giving $[a]_D$. Landolt and other German scientists filter sodium light through a Lippich "ray-filter", (1) potash bichromate, (2) uranium sulphate. This gives a closer approximation to D ray and consequently constants of slightly different value. *Endpoint devices giving shade comparisons.* — Jellet-Cornu ("split") prism, planes of polarization making a slight angle with each other in each half of the field. How used. Disadvantages: Relationship of angle of planes of polarization to sensitiveness, to intensity of light. Laurent's half disc and rocking polarizer. Advantages gained: The most desirable instrument today. Landolt-Lippich polariscope uses small Nicol prism in one-half of field in front of polarizer instead of half-disc of quartz. The Wild "polaristrobometer." End point: Disappearance of interference fringes made by Savart polariscope; polarizer rotates and carries scale; inconvenient but accurate.

EVOLUTION OF THE SACCHARIMETER.

Commercial demand for an instrument giving a direct reading of per cent of sugar in sample without necessity of calculation. Necessary conditions (1) l, v, and w, constants (2) when $w = w'$, $a = 100$ divisions of scale. ∴ (When w = any weight of pure sugar in w' of a sample) $w : w' = $ R (the scale reading) : 100. In ''saccharimeters,'' $l = 2$ decimeters, $v = 100$ cubic centimeters. The fixed value of w' always taken for solution depends on graduation of the scale, and is known as ''normal weight,'' (N). General formula for calculation of per cent when other than the normal weight is used : $\% = \mathrm{R}\dfrac{N}{w'}$.

Saccharimeter Scales. Calculation of N for scale of angular degrees when $l = 2$ and $v = 100$. Objectionable size of weight. Duboscq scale. $a = $ rotation of yellow ray caused by a millimeter section of ''right-handed'' (dextro-rotary) quartz cut perpendicular to the optical axis. N = 16.35g. Same scale used by Laurent. More modern scale, N = 16.19g.

Quartz-wedge Saccharimeters, Inconvenience of saccharimeters with rotating analysers on account of the necessity of monochromatic light. The Soleil quartz-wedge compensator, measures the thickness of a section of ''left-handed'' (lævo-rotary) quartz necessary to neutralize the dextro-rotary action of the sugar solution. *Independent of the source of light, since the dispersive power of quartz and sugar solutions are almost exactly equal.* On this account *the thickness of the quartz section necessary to restore the position of the plane of polarization of any ray bears a constant relation to the concentration of the sugar solution which originally displaced it.* Limitations to sensitiveness of the Soleil-Duboscq saccharimeter, owing to the small size of the normal weight. Ventzke's scale : 100 point, originally the rotation caused by a solution of cane-sugar of 1.100 Sp. Gr. This now taken as equivalent to N = 26.048g. Function of '' sensitive-tint producer.'' Details of instrument much improved by Scheibler and known as Soleil-Ventzke-

Scheibler. This saccharimeter used more than any other in commercial work. Objections: Use prevented by color-blindness. Sensitiveness much decreased by dark, highly-colored solutions. Gradually being superceded by the Schmidt and Haensch "Half-shade" modification of end-point by use of Jellet-Cornu prism. This instrument in use by the Am. Sug. Rfys. Co. and the U. S. Dept. of Agriculture. Latest device gives a triple-shade field by an end-point device on the principle of the Lippich polariscope, —combination of three Nicol prisms—said to have increased sensitiveness.

SOURCES OF ERROR IN "POLARIZATIONS."

(w') weighings to 0.005 gram sufficiently exact. (l) 2 decimeter tubes exact to 0.1 millimeter. (v) 100 cubic-centimeters to 0.05 cubic-centimeter ; —1 cubic-centimeter = volume of 1 gram of water weighed in air at 17.5°C, employed in graduation of all instruments in common use in this country. Recent German saccharimeters standardized for *true* cubic-centimeters, where 1 cc = volume of 1 g of water weighed in vacuo at 4°C.

Errors of Instrument. (1) (Rotary) Eccentricity of axes of scale and rotation. Corrected by readings in opposite quadrants. (2) Distortion of cover-glasses of tubes by pressure — screwing caps too tightly. Practically impossible in tubes as now constructed. Landolt's tubes. Laurent's (3) Errors of graduation calculated for by standard quartz-plate readings and calibration by "control tube." (4) "Zero-error," — plus or minus constant correction. (5) Errors of observation eliminated by averaging results. (6) Variations from standard temperature : Error in most cases, slight. Corrections by tables of Wiley, Andrews, and Mategczek. Effects of temperature variation complicated and obscure.

OTHER COMMON DETERMINATIONS OF SUGAR ANALYSIS.

Moisture. No exact rapid method for drying sugars and syrups known, owing to decomposition. Attempts to obviate this by vacuum-apparatus, drying in current of hydrogen, etc. of doubtful success. Standard Commercial

Method : Soak up dilute solution of syrup by sand and dry at 105°C. till loss is not greater than .2% per hour.   *Ash* obtained in usual manner by ignition in platinum dish at dull-red heat.   Difficult, owing to light bulky coal formed in burning.   Obviated somewhat by admixture of vaselin or pure benzoic acid.   Standard Commercial Method : Treatment with concentrated sulphuric acid before ignition and making an arbitrary deduction of 10% of weight of ash to allow for excess due to formation of sulphates.

"*Reducing Sugars*" by volumetric Fehling method which is sufficiently accurate owing to the relatively small amount of these sugars present.

APPLICATION OF POLARISCOPE DETERMINATIONS IN THE ESTIMATION OF OTHER SACCHARINE SUBSTANCES.

Honey.   Practically pure invert-sugar.   Usually 0.5 to 2% cane-sugar in natural honeys; in rare cases as much as 4%.   Estimated by double polarization.   *Lœvulose*, by Wiley's method based on the change of rotation caused by temperature.   Variation for one gram in 100 cc is .0357° (S. & H.) or .0126°, D per degree C.   Specially devised tubes necessary for reading the solution at a high and low temperature.   Principal adulterations of honey ; commercial glucose and cane-sugar.

Lactose.   Method analagous to cane-sugar determination, using a normal weight proportional to the specific rotatory power of lactose. $N = 32.91$g, when $v = 100$ and $l = 2$ for S. and H. saccharimeter.   Acid-mercuric nitrate is used for a clarifying agent.   Correction must be made for the volume of precipitated casein by method of "*double dilution* :" a = reading when $v = 100$, b, the reading when $v = 200$.   Then, the true reading is $\dfrac{ab}{a - b}$.

ANALYSIS OF COMMERCIAL GLUCOSE.

Viscid, colorless, highly concentrated and refined solution (75–85%) of maltose, dextrose, and dextrin with trace of albuminoids and oil, 0.3 — 0.4% mineral matter.

Three determinations necessary in analysis :   (1) *Total carbohydrates* (anhydrous) by Brix spindle or specific-gravity

apparatus, corrections being made for influence of mineral matter ("ash"). (2) "*Cupric Reducing Power*," (K) = $\dfrac{\text{\textit{copper reduced by} 1 \textit{gram of carbohydrate}}}{\text{\textit{copper reduced by} 1 \textit{gram of dextrose}}}$, by Fehling gravimetric methods : (a) Allihn's method by reducing to copper ; (b) Wiley's modification by electrolysis ; (c) Defren's modification of O'Sullivan's copper-oxide determination.

(3) *Specific rotatory power*: $[a]_D$. Solution must be previously heated to boiling, to obviate possible influence of "birotation." All constants used in calculation and values obtained are referred to a weight of anhydrous carbohydrate calculated from the density of the solution, on the assumption that this density corresponds to a concentration identical with that of a cane-sugar sugar solution of same density. *Calculation of Analytical Results:* If g = % dextrose, m = % maltose, and d = % dextrin :

(1) $g + m + d = 1$.

(2) $g + .61m = K$.

(The cupric reducing power of maltose being .61 that of dextrose.)

(3) $53.5g + 135.2m + 195d = a$.

(These figures giving a of dextrose, maltose, and dextrin respectively.)

$$m = \frac{a + 141.5k - 195}{26.52}$$
$$g = K - .61m$$
$$d = 1.00 - m - g$$

This method gives correct *proportional* amounts of these carbohydrates per unit of anhydrous glucose, not the absolute weights.

ANALYSIS OF WORTS AND MALT SYRUPS WHEN RESULTING FROM ACTION OF DIASTOSE ON STARCH ALONE.

Identical methods, but since maltose and dextrin alone are present :

$$m + d = 1$$
$$.61m = K$$
$$135.2m + 195d = a.$$

Obviously two equations only are necessary.

II. LABORATORY MANIPULATION.

GENERAL NOTES AND DIRECTIONS APPLYING TO SACCHARIMETERS AND POLARISCOPES.
CARE OF EYESIGHT.

Before taking readings, be satisfied that the field is as evenly and brightly illuminated as possible, and the image of the field *sharply* focussed. See that the illuminating apparatus of the scale is in proper order. To adjust focus and illumination in shadow instruments, turn the scale some divisions from zero to get full volume of light. Practice several rapid readings, averaging the results rather than fatigue the eyes by long operations. The shorter readings, if conscientiously made, are more accurate. *End-point.* Move the shadow slightly from one side of the field to the other several times, confining attention to the *central vertical line*. Take the point of *transition* across this line as the end-point. This is theoretically identical with the point of even illumination of the field, but it is easier to locate end-point in the former manner, since often through dust, imperfect adjustment of prism or other defect, poor illumination, uneven dispersion in wedge instruments, etc., it may be impossible to get both halves of the field to look exactly alike. Turn scale-lights off immediately after using, as the glare quickly impairs sensitiveness of vision.

In beginning work with an unfamiliar instrument, set scale to zero and study the changes in the field about this point. This is better than hunting blindly for what you may not recognize, with possible injury to eyesight and instrument. It is especially important when some unusual end-point is observed, as in the Wild polariscope.

CARE OF INSTRUMENT.

Like many instruments of precision, polariscope apparatus is extremely sensitive to derangement from shocks caused by careless handling. Instruments should be disturbed as little as possible, except in the usual manipulation of testing. Nicol prisms from the nature of their material (calc-spar) are peculiarly liable to injury. Calc-spar, being much softer than glass, is easily scratched by careless handling or cleaning. Its peculiar crystallization

makes it liable to split from rapid changes in temperature, or in overheating by placing instrument too close to the lamp. It is easily corroded by acids caused by the fermentation of sugar solutions carelessly spilled in the trough of the instrument. *With proper care* polarizing apparatus will last a lifetime. As the accuracy of the sugar-chemist's work is so dependent on the precision of the instrument, daily practice in such care as will insure this precision is a necessary part of the knowledge and duties required of every worker in a sugar-laboratory. Handle the instrument with clean hands. See that the flame of the lamp is about 200 millimeters from the front end of the instrument, the length of an ordinary polariscope tube. This avoids overheating, which not only endangers the prisms but throws the instrument out of adjustment. With most types, it also insures an evenly illuminated field of maximum brightness. *Only when necessary* clean lenses, quartz-plates and cover-glasses with absolutely clean filter-paper or linen cloth, never with silk or chamois, as the rough surfaces of these fabrics are liable to hold grit. Always rub *very lightly*. In case the edges cannot be reached, remove dirt very carefully with a clean pointed stick of soft wood, as a tooth-pick. Clean Nicol-prisms with especial care, and only when absolutely necessary. In the best instruments, Nicol prisms are enclosed in glass.

POLARISCOPE=TUBES.

Before placing tubes in trough, carefully wipe away all traces of solution or other accidental moisture, and avoid hitting tube against scale or other delicate part. In placing caps on polariscope-tubes, note that the number (or symbol) corresponds with that on the end of the tube. This will insure a tight tube, and prevent jamming or sticking of the caps. All apparatus that has been in contact with solutions should, immediately after using, be washed inside and out in running water and placed in rack to dry. This rule is necessary to insure a constant supply of clean, dry apparatus. Take care to wash the *brass-work* at the ends of the tubes, as this prevents caps from sticking. After washing, leave *cover-glasses* out to facilitate drying; but place *caps*

on ends of tubes, so as to protect ground-glass surfaces from chipping by any accident. The *lead* in sugar solutions gradually clouds glass-ware. Remove by occasional washing with hydrochloric acid. If cover-glasses stick in caps, force them out with a stick of *wood* (*not* of metal or glass.) [See also under *Polarization of Sugar Samples.*]

Scale-Readings.— Always correct readings for *zero-error* (the difference between the end-point observed as read on the scale and the zero of the scale) noting whether this is + or —.

In case zero-error is not large, it is better to allow for it in calculations than to attempt to bring the scale into perfect adjustment.

Take the average of six readings for all exact work, rejecting the *first* reading if it shows much discrepancy from the others.

All saccharimeter scales are read and expressed in percentages and tenths. Rotary polariscopes give readings in degrees and minutes. In *calculations* the minutes are expressed as decimals of a degree. Saccharimeter scales are *divided* into per cent divisions, rotary scales either into halves or thirds of a degree. In both cases the *fractions* of these divisions are determined by "verniers." A vernier (so called from the inventor, a French mathematician) is a double sliding scale extending in both directions from a zero-mark, each half of which has a length in divisions of the main scale *one less* than the number of parts into which it divides the division, but this length of vernier is itself divided into a scale of just the number of parts that the vernier divides the main scale division. For instance, a vernier to divide a scale-division into *ten* equal parts is itself *nine* scale-divisions long and divided into *ten* equal parts. Hence each division of the vernier is $\frac{9}{10}$ of the original scale division. Starting from the *zero* on the vernier, and going *in the direction of the scale reading*, the *number* of the *line of the vernier which coincides with a line on the main scale* gives the number of the parts of the scale-division which the index (zero of vernier) marks. Hence, in the case of a vernier reading tenths, if the zero of the

vernier lies beyond the sixth division and the ninth line of the former coincides with a line of the main scale the reading would be 6.9 divisions. When the zero of the vernier lies on the *minus* side of the scale the lines on the corresponding side of the vernier are read. On rotary scales, verniers usually read to even minutes only, half degree divisions being divided into fifteen parts, thirds of degree divisions into ten parts. In reading rotary scales the number of divisions marked by the zero of the vernier is first read, and then expressed in degrees and *minutes*. *Not until this is done* should the vernier be read and its reading added. Study system of division till you thoroughly understand it before taking readings.

NOTES APPLYING TO SPECIAL INSTRUMENTS.

Schmidt & Haensch Half-Shade Saccharimeter. — $w' = N$ = $26.048g$, when $v = 100$ and $l = 2$. + readings to right. Vernier reads to 0.1%.

Laurent polariscope. — (*Half-Shade.*) Rotary scale, vernier reads to $2'$. Sugar-scale, vernier reads to 0.1%. $w' = N$ when $l = 2$ and $v = 100$ (N to be determined by quartz-plate comparisons with S & H saccharimeter). See that flames of lamp are properly adjusted and that platinum-gauze baskets have their edges parallel to but not touching flame-cones. Add new salt-paste every five or ten minutes. Swing instrument till it points *at* the flames. Turn the analyser off from zero, and focus image of half disc sharply with eye-piece. Turn the analyzer to zero, and *raise* lever on left, behind the scale as high as it will go, or as will let just light enough pass to form a well-defined image. This gives maximum sensitiveness. Determine zero-error at 0 and 180°, when construction of instrument admits (+, in direction of the going of the hands of a clock.) If eccentricity is less than $2'$, readings in the opposite quadrant can be dispensed with. Avoid turning adjustment-pinion on the eye-piece tube, as this throws the prism out of adjustment with the scale. This instrument, adopted by French Government for sugar testing.

Soleil-Duboscq Transition-Tint Saccharimeter. — (+readings to *left*.) Scale shows % of pure sugar when $w' = N$,

$l = 2$, and $v = 100$ (N to be determined by quartz-plate comparisons with S. and H. saccharimeter.) Read to 0.1 % by *estimation*, as the scale has no vernier. See that image is properly focussed by moving eye-piece. (Moves in slot.) Set scale to zero by pinion below the wedges before looking through instrument. Turn *milled head in eye-piece* (which has a movement through *half* a circle) till color is almost white, then adjust color to a *very pale* tint (usually a pearl or a flesh tint) best suited to your eye for a back-ground for showing the pale-rose transition tint which appears in one-half of the field when the scale is not quite at zero. True zero is the point at which *both halves* of the field have the same weak color which changes at the slightest movement of the quartz-wedges, showing *rose* in one-half and its complementary (green) in the other. If the milled head which turns the eye-piece prism ("sensitive tint producer") is adjusted to too strong a tint, the sensitiveness of the instrument is greatly decreased. When a tube containing a solution is placed in the instrument the tint-producer must be readjusted till a sensitive tint is again obtained. Reading by noting transition of tint across central line, as in case of shadow instruments, is recommended. Keep the hood as dark as possible and avoid looking at bright lights before reading, as this causes the eye temporarily to lose its power of distinguishing delicate shades. For same reason avoid fatiguing the eye by looking at the image for more than a *few seconds*. Frequent quick readings give the best results. With most observers readings agree only within 0.3 to 0.5%. On this account this instrument is rarely used now in commercial work.

Soleil-Ventzke-Scheibler Transition-Tint Saccharimeter. A much improved and more sensitive form of transition-tint saccharimeter. The most common commercial instrument in use in America and Germany. Wedges and scale identical with S. and H. saccharimeter ($N = 26.048$g.) Sensitive-tint producer in front of polarizer and worked by milled-head on long spindle at right of eye-piece. Notes on manipulation of Soleil-Duboscq saccharimeter apply to this instrument, which has identical optical parts. Easily reads to 0.1% by vernier.

SUGAR LABORATORY EXERCISES. MASS. INSTITUTE OF TECHNOLOGY.

(15 hour course.)

No.	Determination.	Instrument.	Test Used.
I.	$\dfrac{1^0\text{s \& H}}{1^0\text{D}}$	Laurent (A or C) S & H (B or D)	Quartz-Plate (A or B)
II.	$\dfrac{1^0\text{s \& H}}{1^0\text{D}}$	Laurent (A or C) S & H (B or D)	Any commercial glucose solution of about 10% in 2dm. tube.
III.	N of Laurent.	Laurent (A or C) S & H (B or D)	Any Quartz-Plate.
IV.	N of Soleil-Duboscq.	S-D (E) S & H (B or D)	Any Quartz-Plate.
V.	Polarization of three samples of raw sugar.	*Each* on S & H (B or D)	Any sample of raw sugar (except *second* sugars.)
VI.	Readings of each tube on two instruments.	*Two* on Laurent (A or C)	
VII.	Use *calculated* normal weights. Make up one tube of solution from each sample.	*One* on S-D (E)	
VIII.	$[a]_\text{D 386}$ of Commercial Glucose.	S & H (B or D) (Use data from II. $\frac{p}{100}$ by Brix spindle.)	Any sample of com-glucose diluted to 10% solution.
IX.	$[a]_\text{D}$ of Tartaric Acid. (Plot values, and calculate absolute specific rotatory power roughly.)	Laurent (A) $l = 4$ or 5	C. P. Tartaric Acid three solutions. $c = 5$ $c = 10$ $c = 20$
IX (alternate.)	$[a]_\text{D}$ of Amyl Alcohol.	Laurent (A) $(1 = 4$ or 5)	Com. Amyl Alcohol. (Take sp. gr. by pyknometer.
X.	Double Polarization by Clerget's Method.	S & H (B or D)	Any Molasses or 2nd sugar.
XI.	Calibration of S + H by control-tube.	S & H (B or D) using control-tube.	Granulated sugar.
XII.	Quotient of purity. Calculation of formula for determining % of sugar.	S & H (B or D) " Total Solids " by Brix spindle.	Any 2nd sugar or molasses diluted to about 10%.

The *printed blanks* provided should be filled in duplicate for each determination (12 in all) giving *all data*. The *right* half is for the instructor, the left being retained by the student for filing in his note-book. Calibration tables, plots, etc., can be placed on *back* of blanks.

QUARTZ-PLATE COMPARISONS.

Standard quartz-plates of known rotation are usually employed to determine the values and correctness of the graduation of the scales of instruments. In this laboratory they are used to determine, (1) The equivalent of one division of the S. & H. saccharimeter in angular degrees of rotation of the D ray;* (2) The normal-weight of the Laurent saccharimeter by comparison with the S. & H. scale of known normal-weight (26.048g); (3) The normal weight of the S. D. saccharimeter by comparison with the S. & H. scale.

Take readings as near 20° C as possible. Record *temperature*, so that corrections can be made if necessary. The changes of specific-rotatory power of the quartz-plate from variations of temperature are compensated for by a similar change in the quartz-wedges of the saccharimeter. The other effects of change in temperature are complex and indeterminate, but in most cases of ordinary work negligible, even in rotary instruments unless the variation from 20° is considerable. Quartz-plates should always be placed in the instrument in the position indicated by a mark or stud on the mounting, since they are rarely ground so perfectly as to read the same in every position. [In this laboratory quartz-plates are read with the mark (×) at the *top* of the mounting, the end holding the quartz *facing* the observer.] Many of the recent German quartz-plates read higher than their recorded figures, as they are standardized for instruments with graduations based on a normal weight of 26.048g of sugar in 100 *true* cubic-centimeters, (1 cubic centimeter being the volume occupied by one gram of water, weighed in vacuo at a temperature of 4° C.)

VALUE OF DIVISIONS OF SACCHARIMETER SCALE IN ANGULAR DEGREES BY COMPARISON MADE WITH A TEN PER CENT COMMERCIAL GLUCOSE SOLUTION.

Make comparisons between readings of Laurent polariscope and S. & H. saccharimeter precisely as with quartz-

*All results expressed for "D ray" are obtained by the sodium light filtered through potash bichromate. They differ slightly from those values obtained by the Lippich ray-filter which is said to give a closer approximation to the true D ray. The former method, however, furnishes the light standard generally adopted.

plates, using tube containing a solution of commercial glucose of approximately ten per cent. The value obtained differs slightly from that got with quartz-plate, since the quartz wedges, having a slightly different dispersive power from the glucose, will not restore each rotated ray quite to its original position. This is also shown by the fact that when the glucose solution is read in the saccharimeter, the two halves of the field at the end-point have weak contrasting tints. This determination suggests the limitations of the quartz-wedge saccharimeter in general scientific work, as it can be used only when the dispersion of the solution measured approximates closely to that of quartz. In every case the value of the divisions of the saccharimeter in angular degrees of rotation of yellow light should be determined by comparisons made with solutions of the substance approximately at the concentrations to be used in subsequent investigations, (a) in such cases obviously being the reading of the saccharimeter multiplied by the ratio obtained. The difficulty in reading the end-point owing to the inequality of tint can be obviated by the use of a " *mono-chromatic*" *eye-piece* which contains a section of a potassium bichromate crystal. This absorbs the blue rays, which give the most trouble in reading.

CALIBRATION OF FLASKS.

Instruments in present use are standardized for volumes of 100cc, where one cubic centimeter is taken as the volume occupied by one gram of water, weighed in air at 17.5°C. Hence, all flasks should be calibrated on this standard. Weigh the thoroughly cleaned and dried flask to 0.01g. Weigh again when filled with freshly distilled water of known temperature. The volume of the flask can then be computed from the following formula: $v = P\dfrac{d}{d'}\,[1 + .000025$ $(t - 17.5)]$ where (v) = the required volume, (P) the weight of water in grams up to the mark, at $(t)°$ C, (d) the density of water at 17.5° C and (d') the density at the temperature of weighing. That part of the formula in brackets expresses the effect on the volume of the expansion

of the glass of the flask and is small enough to be omitted in ordinary calibration. The modification of this procedure for double-marked flasks is obvious.

POLARIZATION OF CANE=SUGAR SAMPLES. GENERAL. COMMERCIAL METHOD.

Mix representative sample thoroughly. In these laboratory exercises, weigh 15–20 grams (instead of the normal-weight of the saccharimeter used) as *rapidly as possible,* to avoid change in uniformity of sample by evaporation or drainage. Weigh in tared *German-silver dish* provided for the purpose. Return the remainder of sample *at once* back into covered jar or box. Pour about 50cc of water into dish and grind up crystals with the little *pestle* provided, till they are practically dissolved. Pour solution off into 100*cc calibrated flask*, avoiding pouring out any undissolved sugar. Add a little more water to dissolve the rest of the sugar. In raw sugars there is usually a slight residue of insoluble matter, usually sand or dirt. Wash entire contents of dish into the flask, add about 2cc of *basic lead acetate*, the amount varying with the nature of the sugar, and make up to mark with water.

If foam prevents the reading of the meniscus, add a drop of ether, which will dissipate it. Shake solution up thoroughly and filter through a dry filter into a dry cylinder, rejecting the first few drops. Cover the funnel with a watch-glass to prevent evaporation. The filtrate should be clear, not necessarily colorless. Rinse polariscope-tube twice with the solution, filling half-full, shaking and pouring away, then fill completely. Before placing tube in the instrument wipe carefully, and see that outside of cover-glasses is clean and free from moisture. If objects cannot be seen clearly and without distortion when looking through the tube, it must be refilled. If the tube is handled much, the heat of the hand will sometimes temporarily disturb the solution, which will become clear in a few minutes if the tube is placed in the instrument. Dark solutions, as molasses, sometimes require either diluting before polarizing, the calculation being modified accordingly, or the use of a

one-decimeter tube. Sometimes the solution is decolorized. Place a few grams of prepared bone-black in the dry filter. Reject the first third of the filtrate (first 30cc.)

To highly refined sugars (granulated sugars) add only one cubic centimeter of lead solution, shake and add a few cc of sodium sulphate or chloride to remove excess of lead. A cream of aluminum hydrate, made by precipitating a concentrated potash-alum solution with ammonia and then adding an equal bulk of the alum solution, works very well with very dark solutions, as well as those of highly refined sugars*.

DETERMINATION OF CANE=SUGAR WHEN OTHER OPTI-CALLY=ACTIVE SUBSTANCES ARE PRESENT BY METHOD OF DOUBLE POLARIZATION.

Fundamental Principles. (A) Cane-sugar alone of the more common carbohydrates with which it is associated is hydrolyzed (inverted) by hydrochloric acid when this treatment is carried out according to certain strictly limiting conditions. (B) A definite weight of pure cane-sugar by inversion has its rotation changed by a constant number of divisions of the saccharimeter for a given temperature. Hence the amount of change of rotation of the normal weight of any sample containing cane-sugar will be proportional to the per cent of sugar it contains (i. e. in the same ratio to the amount of change in the normal weight of *pure* sugar, as the sugar percentage of the sample is to 100. (C) As the specific-rotatory power of lævulose, one of the products of the inversion, decreases rapidly as the temperature increases, this change of rotation must be calculated for a standard temperature. (D) As the amount of change of rotation (i.e. the algebraic difference between the polariscope readings before and after inversion) alone is the measure of the cane-sugar present, and since this sugar alone is changed by the process,† the rotatory effects of

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* The bulk of the precipitate formed by clarifying agent is so small as to be negligible) in ordinary polarizations. In exact polarizations of very low grade products the reading should be corrected for by Scheibler's method of Double Dilution [See Lecture Notes : Determination of Lactose.]

†When considerable quantities of commercial glucose are present, a slight correction based on a formula of Weber and McPherson (J. Am. Chem. Soc. 17.319), and amounting to a few tenths of one per cent, is made for the hydrolytic action of the acid on the dextrin present.

other optically-active substances present have no influence on the result. (E) As the concentration of the solution after polarizing "directly" has to be changed by the addition of the inverting acid, the "invert reading" must be corrected accordingly.

CLERGET'S METHOD OF DOUBLE POLARIZATION.*

Prepare solution and polarize in the usual way. Take 50cc of the filtrate prepared for polarizing and measured out in a 50–55cc double graduated flask. Fill to 55 mark with hydrochloric acid, Sp. Gr. 1.20. Heat gradually to 68°C, taking about ten minutes to reach this temperature (not less than ten nor more than fifteen.) [Heat to blood-heat (about 40°C) in naked flame, then place in oven provided for the purpose. Oven temperature should be about 120°C.] Take temperature readings on a thermometer whose bulb is at the centre of the bulb of the flask. When at 68°, cool at once under running water. Wait till solution has reached room temperature and any lead precipitate has subsided, then polarize in a 220 tube provided with a thermometer. Take temperature of the solution at time of polarizing, to 0.1°C.

$$\% = \frac{\text{(direct reading)} - \text{(invert reading)}}{144 - \frac{t}{2}}$$

where (t) is the temperature of the invert-solution. If the normal-weight of the original sample is not used, multiply by $\frac{N}{w'}$. If 200 mm tube is used for polarizing the invert-solution, multiply by $\frac{11}{10}$.

DETERMINATION OF SPECIFIC-ROTATORY POWER.

The specific-rotatory powers of many optically active substances are not strictly constant when calculated from solutions of different concentration. While water solutions of many compounds, as cane-sugar, give values nearly iden-

*This is practically the original method of Clerget, which is considered by the author preferable to the official German modification with which it must not be confounded. The factor of calculation of the latter is somewhat different.

tical when obtained from the different concentrations of
ordinary laboratory practice, there are bodies, as "invert-
sugar" (and its characteristic component, lævulose), or
tartaric acid, showing wide variation. Changes in temper-
ature also often affect the results, particularly in the case of
"invert-sugar" and lævulose.

For practical purposes of general work $[\alpha]_D$ is calculated
from solutions at 20°C containing 10g of substance in 100cc,
its value being taken as a constant. Solutions from which
calculations of specific-rotatory values are to be made are
prepared as nearly as possible at this concentration.

The true ("absolute") specific-rotatory power at any
standard temperature is that when the substance is at the
concentration $C = 1$, independent of the influence of solvents
on the rotation, a condition for polarizing solids reached in
theory only, in most cases. The influence of solvents can
be eliminated and this absolute value calculated with great
exactness by plotting the values of the apparent specific-
rotatory powers calculated at different concentrations, using
abscissæ representing percentages of the active substances.
The plot thus obtained determines the curve of influence
caused by the solvent. By analytic geometry the curve can
be extended till it cuts the ordinate at the 1.00 point. The
value of this ordinate will be the absolute specific-rotatory
power free from the influence of solvents. The accuracy of
this method has been proved in cases where specific-rotatory
values can be determined when free from or under the influ-
ence of different solvents, spirits of turpentine being a case
in point.

Birotation. — Many substances when freshly dissolved
give temporary readings which usually are some simple
multiple or fraction of the constant reading. This tempo-
rary reading varies gradually, approaching in value the true
constant reading, which is reached in a few hours. This
phenomenon is known as "birotation," and is supposed to
be caused by a gradual hydration of the dissolved substance.
It can be prevented by bringing the solution up to a boil, or
adding a very small quantity of ammonium hydrate. The
name "birotation" came from the study of dextrose anhy-

dride, which when freshly dissolved has a specific-rotatory power for the D ray of 105, which gradually decreases till the constant value, 52.8, is reached. It was for a long time supposed that this birotation was peculiar to dextrose anhydride.

The Specific Rotatory Power of Commercial "Glucose." Commercial "Glucose" is a practically colorless, viscid syrup resulting from the hydrolyzing action of acid at a high temperature on starch-paste. It can be resolved into three primary carbohydrates, the proportion of each varying in different samples with the conditions of hydrolysis. As each of these component carbohydrates is optically-active in a different degree, the specific-rotatory power of a sample of commercial glucose becomes a valuable means of determining its quality. As all samples are highly concentrated aqueous solutions (75-85%) a determination of the anhydrous substance is necessary. Direct drying is tedious and inaccurate, and, as the influence of a given concentration of glucose solution on its specific-gravity has not been known exactly till lately, it has become customary to determine an arbitrary specific-rotatory power based on an assumed weight of anhydrous substance, calculated by using the specific gravity factors of cane-sugar. Thus the per cent of anhydrous glucose is taken as the reading of the Brix (Balling) spindle, which in reality only shows true percentage in *cane-sugar* solutions. If these readings are made on solutions of practically one concentration, taken as 10%, the error introduced is practically constant. The specific-rotatory powers thus obtained are proportional to the absolute values by a constant factor, and hence strictly comparable with each other in the same proportion and readily convertible into the latter. The influence of one gram of cane-sugar dissolved in 100 cubic-centimeters of solution is expresed by the factor, .00386. Hence the weight of cane-sugar dissolved in 100 cubic-centimeters of solution is $w = \dfrac{d-1}{.00386}$. On this account specific-rotatory powers figured on cane-sugar factors are written $[a]_{D386}$ to distinqnish them from absolute values. (Note that this

calculation gives grams in 100 *cubic-centimeters*, the Brix giving grams in 100 *grams*.)

$[a]_{D386}$ *of Commercial Glucose.—Method.* Dissolve about 50g [estimate without weighing] in eight or ten times its bulk of water. Filter through two or more thicknesses of paper to remove any cloudiness. (Lead solution cannot be used for clarifying, as it precipitates dextrin). Determine Brix reading of solutions which should be about 10% (8-12%.) Fill two-decimeter tube, and take reading in a quartz-wedge saccharimeter, calculating (a) by use of the factor obtained in determining the value of one division of the saccharimeter in angular degrees of yellow light rotation measured by glucose.

Brix-Readings. Brix spindles, like all correctly made hydrometers, should be read along a line lying in the *plane of the surface* of the liquid and *not* at line of contact of the liquid with the stem. All readings not made at the temperature at which the instrument was graduated, obviously, must be calculated to this temperature. Use table of temperature corrections. The original Brix spindles were made for a temperature of 17.5°C. Those used in present commercial work are more often made for 15°C or 60°Fh.

Error due to dissolved mineral matter. — In a refined glucose, such as used in commerce, the organic matter other than carbohydrate is so small as to be negligible. The soluble mineral substances is often sufficient to affect the density of the solution to the third decimal place. Hence in accurate work allowance must be made for this source of error. This is done by determining the *ash* of the glucose, calculating the amount in 100 cc of the solution whose density is to be determined, and multiplying this value by .008, which closely represents the influence on the density of one gram of the mineral salts present in 100cc of solution. The density obtained after subtracting this correction represents that caused by solution of the carbohydrates alone.

Determination of Ash of Commercial Glucose. — Take 10cc of solution of known density. Evaporate (practically) to dryness in a platinum dish. Add a small lump of vase-

line (a *very* small lump.) Incinerate by placing dish in a larger dish of platinum or nickel. This prevents over-heating and subsequent loss of chlorides. Protect from draughts, and heat very gradually at first. Before placing dish containing ash in a dessicator, cover with a watch-glass.

CALIBRATION OF SACCHARIMETER SCALE BY THE CONTROL=TUBE.

The correctness of the saccharimetric scale can be established at a few points by comparisons with standard quartz-plates giving readings at these points. If the surfaces of the quartz-wedges are not perfect planes, it is clear that such comparisons do not suffice to standardize the instrument, as they do not provide for the possibility of slight irregularities of surface. It will be noted that an irregularity no greater than .0016 of a millimeter is sufficient to cause an error of 0.1%. By means of the " control-tube " comparisons can be made at any point of the scale, using a solution of pure cane-sugar at three or four concentrations. The control-tube is capable of variations in length through a range of about 100 millimeters,* its length at any position being measured by a scale reading to 0.1 millimeter.

Since the reading (R) of the saccharimeter gives the per cent of sugar $\left(\dfrac{w}{w'}\right)$ in the sample when $w' = N$, $l = 2$, and $v = 100$, the equation for the per cent of sugar at *any* length of tube under these conditions is $\% = R\dfrac{2}{l}$, and for the reading, therefore, $R = \%\dfrac{l}{2}$. If the normal weight of chemically-pure sugar is used, $R = \dfrac{100l}{2}$.

By this means the *actual* reading of the scale at any given length of the control-tube can be compared with the reading *calculated* for that length. In exact work it is customary to make up solutions of chemically-pure cane-

*A form of control-tube is made with an inner cover-glass so that l can be reduced to short lengths, even down to 0. The error introduced in determining these very short lengths is obviously so increased as to make this modification undesirable.

sugar which can be prepared by washing the finely pulverized granulated sugar with two or three times its bulk of 85% alcohol, drying at 105° C, pulverizing, and repeating the whole process. Sugar thus treated gives a reading of 100 when polarized. [In this laboratory, where the exercise with the control-tube is for practice in manipulation, two solutions of ordinary granulated sugar are made up, one 20g in 100cc, the other 10g in 100cc. Readings are taken of each solution at six different tube-lengths, the per cent of sugar in the granulated sample being first determined by calculation from the six readings on the 20g solution. This gives a value which, while it averages the errors of these six points, is a result exact enough for the purpose, since the average error of a first-class saccharimeter is exceedingly small. Calculate the readings at each tube length, using the percentage of sugar calculated. ($R = \dfrac{N\,l}{w'}\dfrac{1}{2}\%$). Make a comparison table on *back* of printed blank with columns arranged as follows:

%	w'	l.	R (actual)	R (calculated)	Dif.,

filling in printed blank with other data as usual.]

Manipulation of Control-Tube. — Insert funnel-plug and fill tube just as if of ordinary type. Remove plug and shorten tube by moving the pinion very slightly. This will make the solution fill up the plug-hole. Insert funnel and fill about half full of solution, taking care not to let air into the main tube. It is advisable to pour a few drops of sugar-solution through the funnel before placing the latter in the tube, as this insures displacement of air in the neck which may otherwise be forced into the tube. The pinion should work stiffly enough to avoid changing the length of the control-tube in handling. Owing to the absorption of the packing of the telescope joint, the control-tube must be much more thoroughly rinsed with the solution than an ordinary tube, especially in changing solutions. Obviously, too, the tube should be very thoroughly washed and dried after using. The vented cap should be used on the funnel to avoid evaporation.

QUOTIENT OF PURITY ("COEFFICIENT"—"EXPONENT.")

This is $\dfrac{\text{per cent sugar}}{\text{per cent solids}}$: theoretically, the per cent of sugar
in the anhydrous substance. In commercial work, the Brix
reading is taken as the per cent of total solids, on the
assumption that the specific-gravity of the impurities of
cane-sugar solutions of commerce is the same as that of an
equivalent amount of cane-sugar. The error is not a great
one in most cases of cane-sugar work. Cane-juices are
determined directly after standing a short time to allow
escape of air. Syrups, molasses, massecuites, etc., are
diluted to about 10°, Brix.

While this determination, based on an assumed factor,
does not give absolute results, it is a rapid means of valu-
ation of sugar liquors on a constant basis of comparison
independent of concentration. It is of great service in
measuring the efficiency of much work of the sugar-house
and refinery.

General Directions. Make up sample into a solution of
a density corresponding to 10-15% Brix. Make a Brix
determination. Find corresponding density in the table.
Fill a 50-55cc flask to 50 mark; add 2-5cc of lead solution,
gauging the amonnt according to the impurity of the sample.
Fill up flask with water to 55 mark. Filter and polarize in
the ordinary manner. (1) Density (d) of the solution;
(2) Reading (R) of the saccharimeter; (3) Its normal-
weight (N); (4) the allowance for change in volume caused
by introduction of the clarifying agent: are the only data
necessary for determining the *per cent of sugar* in the
solution as originally made up, and hence by use of the
Brix determination (B) the quotient of purity (Q) can be
calculated. [Give a formula in terms of R, d, N and B for
Quotient of Purity (Q)]. This method is used in commer-
cial work, as it obviates the inconvenience of weighing the
solution on a balance, as well as the error introduced by the
low saccharimeter readings of the normal weight of so dilute
a solution. A table calculated from the formula desired has
been worked out by Schmitz, by which the per cent of sugar
can be determined by inspection when the polarization and

Brix readings are made in the manner described. This table has been corrected for the change in specific-rotatory power of cane-sugar at low concentrations. In very dilute solutions this affects the third significant figure.

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TABLES.

TABLE A.

BRIX AND SPECIFIC–GRAVITY (8 TO 15° BRIX).

Brix	Sp. Gr.	Brix	Sp. Gr.
8.0	1.03187	10.0	1.04014
1	3228	1	4055
2	3270	2	4097
3	3311	3	4139
4	3352	4	4180
5	3393	5	4222
6	3434	6	4264
7	3475	7	4306
8	3517	8	4348
9	3558	9	4390
9.0	1.03599	11.0	1.04431
1	3640	1	4473
2	3682	2	4515
3	3723	3	4557
4	3765	4	4599
5	3806	5	4641
6	3848	6	4683
7	3889	7	4726
8	3931	8	4768
9	3972	9	4810

Brix	Sq. Gr.	Brix	Sq. Gr.
12.0	1.04852	14.0	1.05703
1	4894	1	5746
2	4937	2	5789
3	4979	3	5831
4	5021	4	5874
5	5064	5	5917
6	5106	6	5960
7	5149	7	6003
8	5191	8	6047
9	5233	9	6090
13.0	1.05276	15.0	1.06133
1	5318		
2	5361		
3	5404		
4	5446		
5	5489		
6	5532		
7	5574		
8	5617		
9	5660		

TABLE B.

TEMPERATURE CORRECTIONS FOR BRIX READINGS.

Degrees C varying from Standard.	Corrections in Degrees, Brix for		
	10°	15°	20°
	Concentrations.		
Below Standard. 7	.28	.31	.34
Below Standard. 6	.24	.26	.29
Below Standard. 5	.21	.23	.24
Below Standard. 4	.18	.19	.20
Below Standard. 3	.14	.16	.16
Below Standard. 2	.10	.12	.12
Below Standard. 1	.06	.06	.07
Above Standard. 1	.06	.06	.06
Above Standard. 2	.12	.13	.13
Above Standard. 3	.19	.21	.21
Above Standard. 4	.26	.28	.28
Above Standard. 5	.32	.34	.35
Above Standard. 6	.38	.40	.41
Above Standard. 7	.44	.46	.48
Above Standard. 8	.51	.53	.55
Above Standard. 9	.58	.60	.62
Above Standard. 10	.65	.68	.69

TABLE C.
DENSITY OF WATER.

t	d	t	d
10	.99974	20	.99827
11	965	21	806
12	957	22	785
13	943	23	762
14	930	24	739
15	.99915	25	.99714
15.5	(.99907)	26	681
16	900	27	654
17	884	28	626
17.5	(.99875)	29	597
18	866	30	.99567
19	847		

SPECIFIC-ROTATORY POWER OF QUARTZ.

As determined by Laurent Polariscope (sodium flame passing through potash-bichromate) for a section 1 mm thick at 20°: $[a]_D = 21.68°$ $[1 + .000179 (t - 20)]$. As determined for more strictly monochromatic yellow light (D ray of spectrum), by passage of light of sodium flame through Lippich "ray-filter" (potash-bichromate and uranium sulphate) for a section 1 mm thick at 20°, $[a]_D = 21.72°$.

CLARIFYING SOLUTIONS.

Basic Lead Acetate. — Boil for half an hour 440 grams of lead acetate with 264 grams of litharge in 1500 cc of water. Cool, and dilute to two litres. Allow to subside, and siphon off clear liquor. (Sp. gr. about 1.27, containing about 35% of the basic salt.)

Alumina-Cream Mixture. — Make a saturated solution of potash-alum. Divide into two parts. Neutralize one part with strongest ammonic hydrate. Add balance of potash-alum solution. For decolorizing dark solutions, and removing excess of lead.

SALT-PASTE FOR SODIUM-FLAME LAMP.
(Modified from recipe of Dupont.)

Ordinary effloresced sodium hydrogen phosphate, three parts. Table salt, two parts. Sodium carbonate, one part. Pulverize, and mix intimately. Make into a stiff paste with glycerine.